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UNIVERSITY OF LOUISVILLE

SEMI-COMMERCIAL DISTILLATION OF TERNARY MIXTURES

PAPER I

GLYCERIN-ETHYL ALCOHOL-WATER

A Dissertation

Submitted to the Faculty

of the

Graduate School of the University of Louisville

In Partial Fulfillment of the

Requirements for the Degree

Of Master of Science in

Chemical Engineering

DEPARTMENT OF CHEMICAL ENGINEERING

by

ROCHUS CORNELIUS STAHL

1936

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The Author Wishes to Express
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Given by Dr. R. C. Ernst
Who Directed this Research

CHAPTER I

INTRODUCTION

INTRODUCTION

The object of this research is the preliminary study of the distillation of a ternary system in a semi-commercial still. Ethyl alcohol, glycerin and water were the materials used in this investigation.

The semi-plant still was operated at total reflux and varying feed composition.

The study of this system in an Othmer distillation apparatus was carried on in this laboratory by Watkins (1). The results obtained in this investigation are to be compared with those of Watkins.

This investigation covers the distillation of the ternary system glycerin-ethyl alcohol-water at total reflux and varying conditions of feed.

1

CHAPTER II

HISTORICAL

HISTORICAL

Distillation is perhaps the oldest art practiced by man. It is evident from the results obtained that the early artisans understood, at least in part, many of the laws governing distillation.

In 350 B. C. Aristotle separated salt from water by distillation.

Dioscorides, in the first century A. D., recovered several fractions from the charge by placing flock wool in the vapor stream over boiling liquid. The lighter fractions condensed on the cooler wool and were recovered by wringing.

The alchemists were able to draw as many as six fractions from a still. They utilized the principles of refluxing, control of still head temperatures, reboiling of condensates, and continuous feeding of the still with preheated liquid.

The first petroleum and coal tar stills were set up in the United States about 1850. These

early stills were of the "cheese box" design. Continuous feeding was first employed about 1885.

The latest design of petroleum stills is the tube and cracking still.

France, the country of famous wines and liquors, has done much of the development of distillation apparatus, especially noteworthy is the development of the modern fractionating column(2).

The mechanism of fractionation of binary mixtures and the design of rectifying columns for the mixtures have been studied by several investigators, among whom are Souders and Brown (3), Chillas and Weir (4), and McCabe and Thiele (5). The method of McCabe and Thiele is the most important because it is simplest and most easily applied. There is no method similar to that of McCabe and Thiele for the calculation of columns for mixtures of more than two components.

Thiele and Geddes (6) and Piron (7) have investigated hydrocarbon mixtures and have proposed formulas for calculation of column but these equations are cumbersome and difficult to use because of the complexity of the mixture and the number of as-

sumptions and approximations that must be made.

Underwood (8), Goodliffe (9), Branden (10), and Lewis and Wilde (11) have written useful papers on the theory and practice of testing stills.

The effect of vapor velocity on entrainment and plate efficiency in the rectification of binary mixtures has been investigated by Carey, Griswold, Lewis and McAdams (12).

The greatest distilling industry, crude petroleum, is not interested in separating the mixture into its components but into "cuts" that have properties which lie in a specified range. This fact explains the small amount of theoretical work that has been done in petroleum distillation.

In the alcohol industry the ternary system ethanol-water-fusel oil must be dealt with. Alcohol and water, and alcohol and fusel oil are miscible in all proportions, but fusel oil and water are only slightly soluble in each other. When this system is subjected to distillation, alcohol with small quantities of water passes to the receiver while the less volatile fusel oil and water remain in the kettle. Since fusel oil and water are

immiscible, steam distillation takes place and carries the fusel oil up the column. The fusel oil with small amounts of alcohol and water are withdrawn near the center of the column. This product is allowed to settle into two layers, an alcohol-water strata and an alcohol-fusel oil layer. The alcohol-water layer is returned to the column while the alcohol-fusel oil mixture is taken to another still for separation. In general the number of columns required to separate a mixture into its components is one less than the number of components.

The physical properties of the system acetone-n-butanol-water have been determined by Ernst, Litkenhous and Spanyer (13). A liquid-vapor equilibrium diagram for this system have been prepared by Neiman (14). This system is similar to the system ethyl alcohol-water-fusel oil in properties of decreasing volatility, increasing boiling points, and immiscibility of water and butanol.

The system under study, ethyl alcohol-water-glycerin is similar to the two previously mentioned systems in regard to decreasing volatility and increasing boiling points, but it does

not have any immiscible components. The constituents ethyl-alcohol, water, and glycerin are miscible in all proportions at low temperatures.

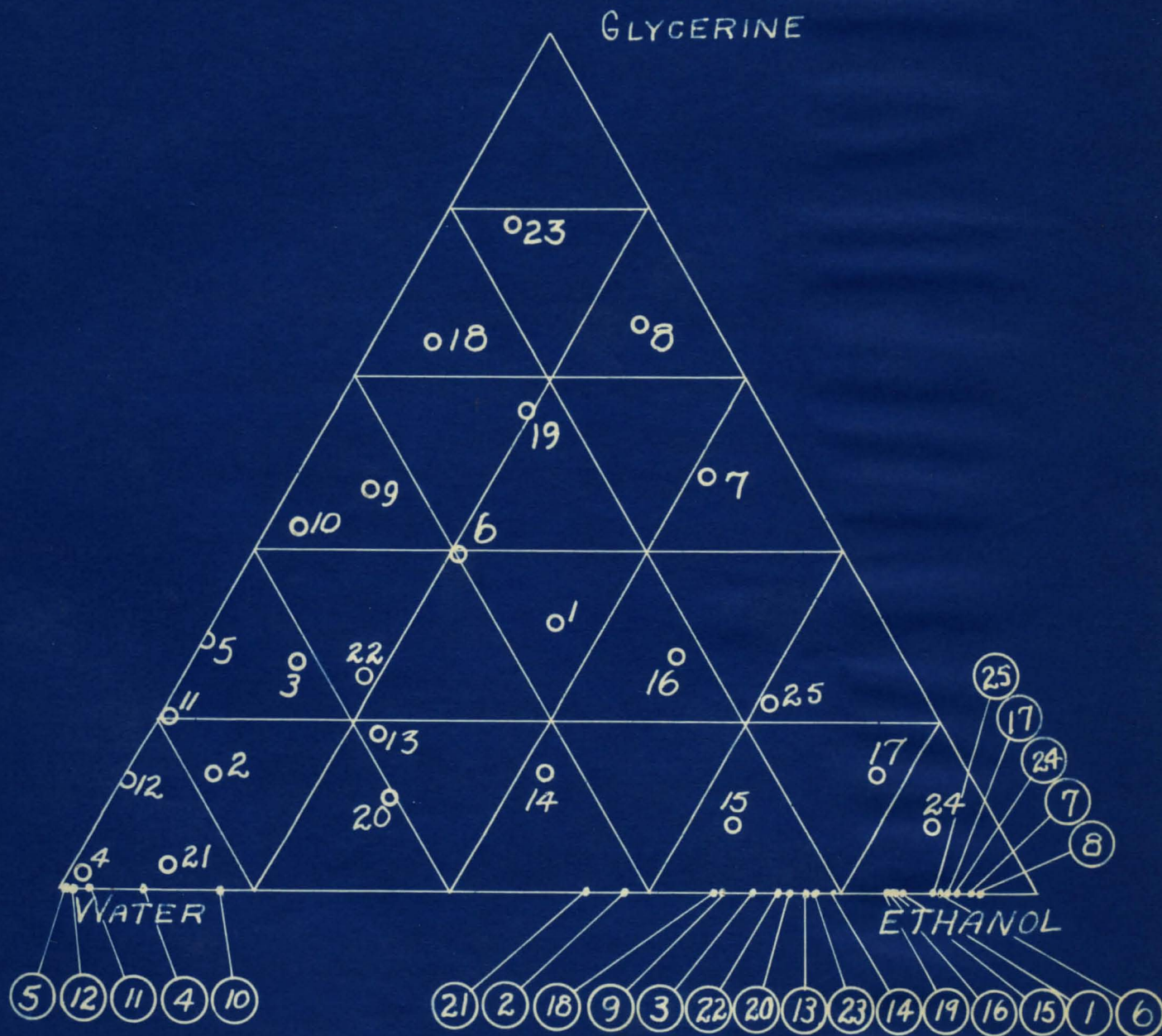
The physical properties of the system ethyl alcohol-water-glycerin have been determined by Watkins (15) and a vapor equilibrium diagram for this system has also been prepared by Watkins (1). See Fig. 1.

Craven (16) has determined the relative volatility of the following systems in the absence of water and at high dilution of water: methyl alcohol-ethyl alcohol; acetone-methyl alcohol; ethyl alcohol benzene; and ethyl alcohol-acetone.

Water is the most familiar of the three components. Its physical and chemical properties have been thoroughly investigated.

Ethyl alcohol has been known since biblical times because of its occurrence in liquors. Because of the commercial importance of ethyl

FIG. 11
 VAPOR LIQUID EQUILIBRIUM
 COMPOSITION DATA
 ○ LIQUID PHASE
 • VAPOR PHASE



alcohol its properties have been extensively investigated. Ethyl alcohol may be prepared commercially by fermentation of starch containing substances such as cereal, potatoes, sugar or cane molasses. In chemical industry ethyl alcohol is used extensively as a solvent, a fuel, and as a raw material for the preparation of other compounds.

Glycerin may be prepared by the fermentation of carbohydrates or as a by-product of the soap industry. Glycerol is used commercially to control moisture in tobacco, and to prepare other chemical compounds. Glycerin also has numerous miscellaneous uses. Because of its broad utility the physical and chemical properties of these compounds are well known.

CHAPTER III

THEORETICAL

THEORETICAL

Distillation is defined as the separation of the components of a liquid mixture by partial vaporization of the mixture and separate recovery of the vapor and residual liquid. The vapor will contain the more volatile components of the mixture in increased proportions. The residual liquid will contain the less volatile constituents in increased concentrations. In order that separation can take place, the vapor composition must be different from the composition of the liquid from which it is formed. Theoretically distillation cannot yield a component in absolutely pure form. The degree of separation depends upon the properties of the components involved, the type of still used, and the operating conditions of that still.

The basic laws of distillation are those which govern the equilibrium of the vapor and liquid phases of the system to be distilled. The more important of these laws are those of Raoult and Henry.

According to Raoult's law, the partial pressure of the solvent is equal to the vapor pressure of the pure solvent multiplied by the mol fraction of solvent present.

Henry's law states that the partial pressure of the solute is equal to a constant times the mol fraction of solute present.

In ideal solutions Raoult's and Henry's laws are identical because there is no distinction between the solute and the solvent and the constant in Henry's law becomes the vapor pressure of the pure solute.

Both of these laws hold in ideal solutions at all concentrations and in non-ideal dilute solutions, but neither holds accurately in non-ideal, concentrated solutions.

Liquids whose properties, when mixed, are additive may be considered ideal and to follow those laws. Benzene and toluene give a solution which is practically ideal.

Two very useful diagrams used in distillation problems are the boiling-point diagram

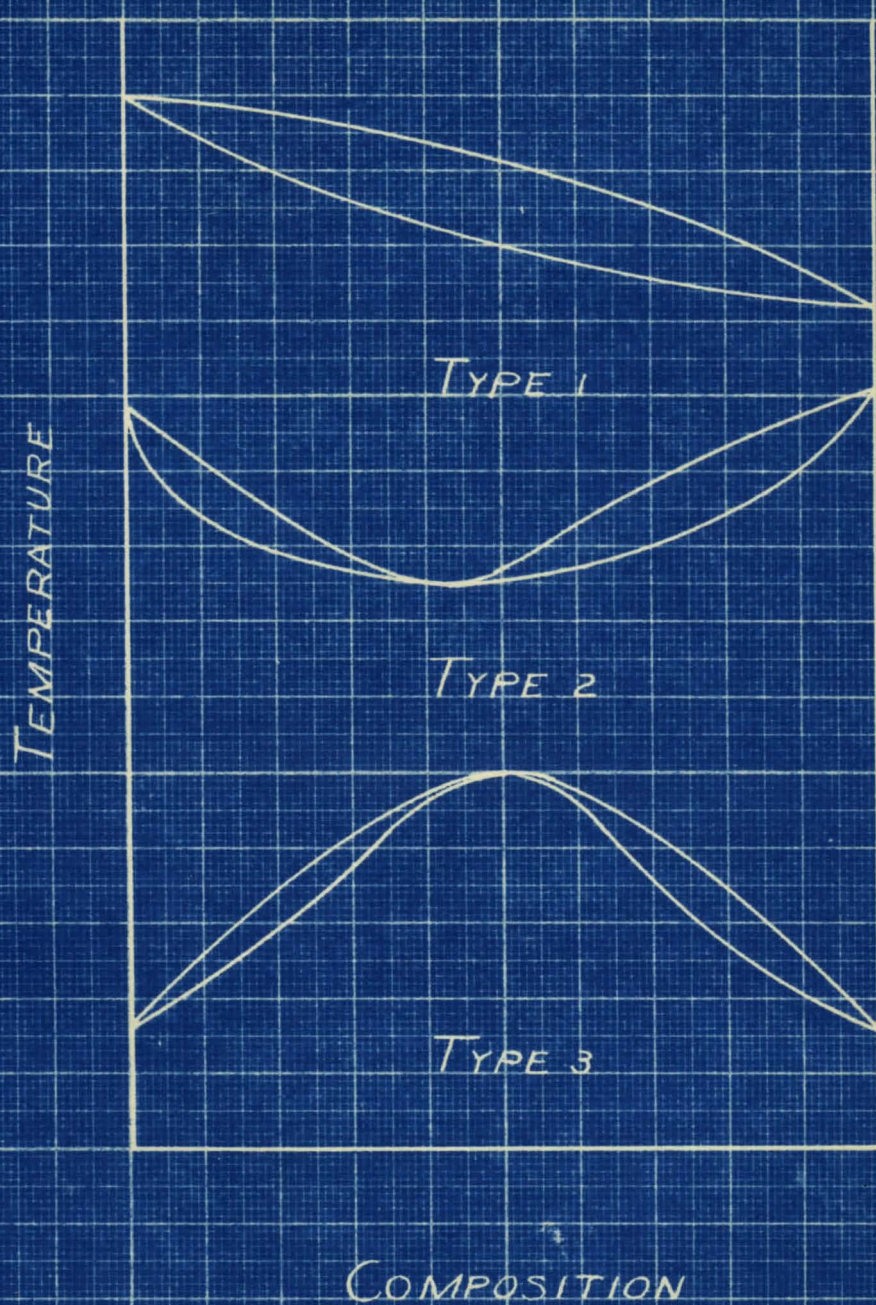
and the vapor-liquid equilibrium diagram.

A boiling point diagram is constructed by plotting temperatures as ordinates and the compositions as abscissas. The diagram will consist of two curves, the ends of which coincide. This is necessarily true because at a given temperature the vapor will not have the same composition as the liquid except in the case of an azeotropic mixture.

When mixtures are boiled at constant pressure, three types of boiling point diagrams occur. See Fig. 2. In Type (1) the boiling point of all possible mixtures are intermediate between those of the two components. All mixtures of Type (2) shows a minimum in their boiling point curve. The boiling point diagram of the system benzene-ethyl alcohol is an example of this type. Type (3) mixtures exhibit a maximum. (The system ethyl alcohol-water.)

The equilibrium diagram is constructed by plotting composition of the vapor as ordinates and composition of the liquid in equilibrium with that vapor as abscissas. This diagram gives a simple relationship between the liquid and vapor in equilibrium with that vapor. This diagram is

FIG. 2
TYPES OF BOILING POINT DIAGRAMS



used in the method of McCabe and Thiele for calculating the number of plates in a rectifying column.

The general gas law and Dalton's Law are used in design calculations to calculate vapor volumes and partial pressures respectively.

The general gas law states that

$$PV = n RT$$

where

P = total pressure

V = the volume of the gas

n = the number of mols of the gas

R = the universal gas constant in
consistent units

T = the absolute temperature

Dalton's law of partial pressures is that the total pressure of a mixture of two or more gases is equal to the sum of the pressures which each gas would exert if it were alone in the volume occupied by the mixture. Expressed mathematically

$$PV = V (p_1 + p_2 + p_3 + \dots)$$

where the partial pressures of the constituent gas are denoted by p_1 , p_2 , p_3 , etc. and P and V

represent the total pressure and volume of the gaseous mixture.

Commercial distillation may be accomplished by boiling the mixture to be separated and condensing the vapor without allowing any of the condensed vapor to return to the still as reflux or by returning part of the condensate to the still column as reflux in such a way that intimate contact is made between the reflux and the vapor.

Distillation without reflux may be carried out by either flash distillation or differential distillation. Flash distillation consists of vaporizing a definite portion of the liquid mixture, usually under pressure, allowing the vapor to remain in contact with the liquid so that equilibrium is maintained. At the end of the operation the vapor is withdrawn and condensed. Flash or equilibrium distillation is of importance only in petroleum refining practice.

In differential distillation the vapor is removed from contact with the liquid and condensed as quickly as it is generated. Most laboratory distillations that do not involve the use of reflux

columns are differential distillations. The old type "topping" petroleum still is an example of this type of distillation.

Rectification consists of bringing a stream of vapor and liquid into intimate counter-current contact in a fractionating column. Rectification may be either a batch or a continuous process. Batch rectification approaches continuous distillation when the quantity of liquid in the still kettle is large compared with the quantity of liquid which has passed to the receiver.

Fractionating columns may be classified as: (1) packed columns, (2) sieve plate columns, (3) bubble-cap columns.

In this paper the term reflux ratio will be used to signify the ratio of the liquid reflux to the rising vapor at any point in the column.

The completeness of separation by rectification depends upon:

- A. Volatility of the components of the system
- B. Reflux ratio

- C. Efficiency of contact between vapor and liquid
- D. Number of plates and the distance between plates and in the case of a packed tower the height of the column.

Plate efficiency is defined by Murphree (17) as

$$E = \frac{100 (y_n - y_{n+1})}{y_n^* - y_{n+1}}$$

where

E = plate efficiency

y_n = composition of vapor leaving the plate

y_n^* = composition of vapor in equilibrium with the liquid leaving the plate.

y_{n+1} = composition of the vapor rising to the plate.

Steam distillation is a distillation in which vaporization of the volatile constituents of a batch of material is effected at a lowered temperature by introduction of a chemically inert gas (usually steam) directly into the feed. This method is used to distill materials that have a relatively high boiling point or that are liable

to decompose at this temperature. This method can be applied only when the product is almost or completely immiscible with the inert gas employed.

In the literature one finds very little data or discussion of the distillation of systems of more than two components. Some systems apparently follow no definite law (the system ethyl alcohol-fusel oil-water) while others (petroleum) because of their complexity defy the proposing of any theory regarding their behavior. There is no method for the calculating of fractionating columns for multi-component systems that corresponds with the method of McCabe and Thiele (5) for problems of binary systems.

In this research the system ethyl alcohol-glycerin-water will be distilled in an eight plate, sieve tower still. The conditions will be varying composition of feed, total reflux, and batch rectification.

CHAPTER IV

MATERIALS

MATERIALS

The ethyl alcohol used was a technical grade containing 94% ethyl alcohol, (Density at 25° C. 0.8030).

The glycerin utilized was also a technical grade analyzing 95% glycerin, (Refractive Index at 25° C. 1.4630).

The tap water employed had a refractive index of 1.3334 at 25° C.

These compounds are miscible in all proportions at low temperatures.

The density and refractive index of the system, data of Watkins (15) are given in figure 3.

Physical properties of the pure components are given in table 1.

This grade of materials gives a close approximation to mixtures that would be encountered in commercial practice.

FIG. 3

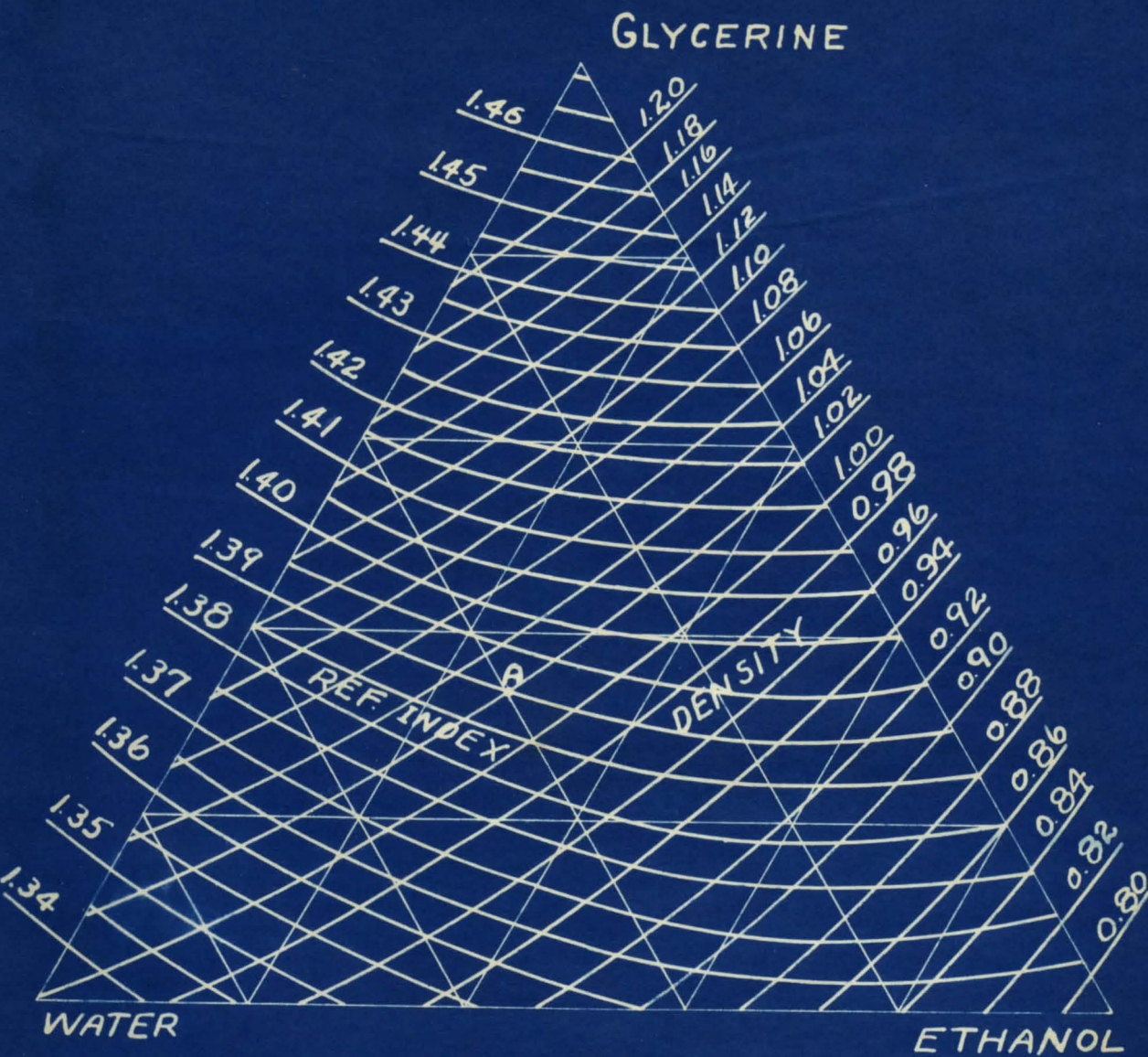


TABLE I

PHYSICAL PROPERTIES OF PURE MATERIALS

| Liquid | Formula | Density | Refractive Index | Specific Heat | Boiling Point |
|---------------|----------------|---------|---------------------|------------------|------------------|
| Ethyl alcohol | C_2H_5OH | 0.7851* | 1.3596* | 0.536* | 78.4° C. |
| Glycerin | $C_3H_5(OH)_3$ | 1.2580* | 1.4729* | 0.555* | 290.0° C. |
| Water | HOH | 0.9971 | 1.3332* | 1.000* | 100.0° C. |

* Data of Watkins (1), (15)

CHAPTER V

APPARATUS



Fig. 4

PHOTOGRAPH OF THE STILL

APPARATUS

The still (see fig. 4) used in this investigation consisted of a still kettle, a sieve plate column, and a reflux condenser.

The copper steam jacketed kettle had a capacity of approximately eight gallons. A discharge valve was located at the bottom of the kettle.

The eight plate sieve type column had an inside diameter of six inches. The distance between plates was six and one half inches. Each plate had a tap for securing samples of the liquid on that plate.

The reflux condenser consisted of approximately two feet of one half inch copper tubing. The cooling water line of the condenser contained taps for taking water samples for temperature reading, and a cold water meter.

An Abbe refractometer was used to determine the refractive index of the samples for identification purposes.

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The density of the samples was found by means of a Westphal balance.

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CHAPTER VI

PROCEDURE

PROCEDURE

The still was charged with twenty pounds of feed of the desired composition. Water was then run at the maximum rate thru the reflux condenser. Immediately steam was allowed to pass into the kettle jacket. The apparatus was then allowed to come to equilibrium. (An indication of equilibrium was a constant temperature rise in the water from the reflux condenser.) The time required for equilibrium was about twenty minutes. Samples of approximately thirty cubic centimeters were taken from each plate and from the kettle. The temperature of each sample was adjusted to 25° C. The density and refractive index of the samples was determined. The composition of the samples was read from the chart. (fig. 3)

Figure 3 is a three component diagram consisting of constant property lines. The composition of a sample is readily ascertained from this chart. As an example a sample was found to have a density at 25° C. of 1.020 and a refractive index at this temperature of 1.390. The composition of this sample is indicated by point A which corresponds to 40.0% water, 27.0% ethyl alcohol, and 33.0% glycerin.

CHAPTER VII

DATA and RESULTS

DATA and RESULTS

The data of eight representative runs are given in the following pages of this paper. All charges contained the three components. The percent glycerin in the feed varied from 10.2 to 60.0. The ethyl alcohol composition ranged from 9.8 to 78.0 percent. Water in the feed varied from 10.0 to 80 percent.

TABLE 2

RUN 1

| Sample | Refractive Index | Density | Water | Composition (percent) | |
|-------------|---------------------|---------|-------|-----------------------|---------------|
| | | | | Glycerin | Ethyl-Alcohol |
| Feed | 1.4210 | 1.095 | 20.5 | 60.0 | 19.5 |
| Bottoms | 1.4402 | 1.199 | 21.0 | 77.5 | 1.5 |
| Plate No. 1 | 1.3534 | 0.954 | 67.5 | 1.5 | 31.0 |
| Plate No. 2 | 1.3626 | 0.883 | 36.0 | 0.0 | 64.0 |
| Plate No. 3 | 1.3637 | 0.848 | 23.0 | 0.0 | 77.0 |
| Plate No. 4 | 1.3634 | 0.834 | 17.0 | 0.0 | 83.0 |
| Plate No. 5 | 1.3634 | 0.827 | 14.5 | 0.0 | 85.5 |
| Plate No. 6 | 1.3632 | 0.820 | 12.0 | 0.0 | 88.0 |
| Plate No. 7 | 1.3630 | 0.816 | 10.5 | 0.0 | 89.5 |
| Plate No. 8 | 1.3630 | 0.812 | 8.5 | 0.0 | 91.5 |

TABLE 3

RUN 2

Composition (Percent)

| Sample | Refractive Index | Density | Water | Glycerin | Ethyl-Alcohol |
|-------------|------------------|---------|-------|----------|---------------|
| Feed | 1.3930 | 1.049 | 41.0 | 39.0 | 20.0 |
| Bottoms | 1.4075 | 1.140 | 44.0 | 55.0 | 1.0 |
| Plate No. 1 | 1.3368 | 0.996 | 94.0 | 0.5 | 5.5 |
| Plate No. 2 | 1.3350 | 0.994 | 94.0 | 0.0 | 6.0 |
| Plate No. 3 | 1.3523 | 0.952 | 68.5 | 0.0 | 31.5 |
| Plate No. 4 | 1.3631 | 0.875 | 33.0 | 0.0 | 67.0 |
| Plate No. 5 | 1.3639 | 0.843 | 21.0 | 0.0 | 79.0 |
| Plate No. 6 | 1.3633 | 0.828 | 15.5 | 0.0 | 84.5 |
| Plate No. 7 | 1.3632 | 0.820 | 12.0 | 0.0 | 88.0 |
| Plate No. 8 | 1.3631 | 0.815 | 10.0 | 0.0 | 90.0 |

TABLE 4

RUN3

| Sample | Refractive Index | Density | Water | Composition (Percent) | |
|-------------|------------------|---------|-------|-----------------------|---------------|
| | | | | Glycerin | Ethyl-Alcohol |
| Feed | 1.4012 | 1.002 | 22.0 | 40.0 | 38.0 |
| Bottoms | 1.4425 | 1.202 | 20.0 | 77.5 | 2.5 |
| Plate No. 1 | 1.3570 | 0.934 | 55.5 | 2.5 | 42.0 |
| Plate No. 2 | 1.3632 | 0.862 | 39.0 | 0.0 | 71.0 |
| Plate No. 3 | 1.3633 | 0.839 | 19.0 | 0.0 | 81.0 |
| Plate No. 4 | 1.3633 | 0.830 | 16.0 | 0.0 | 84.0 |
| Plate No. 5 | 1.3634 | 0.823 | 13.0 | 0.0 | 87.0 |
| Plate No. 6 | 1.3628 | 0.818 | 11.5 | 0.0 | 88.5 |
| Plate No. 7 | 1.3626 | 0.814 | 9.5 | 0.0 | 90.5 |
| Plate No. 8 | 1.3623 | 0.811 | 8.5 | 0.0 | 91.5 |

TABLE 5

RUN 4

| Sample | Refractive Index | Density | Composition (Percent) | | |
|-------------|---------------------|---------|-----------------------|----------|---------------|
| | | | Water | Glycerin | Ethyl-Alcohol |
| Feed | 1.3706 | 1.014 | 60.8 | 20.5 | 18.7 |
| Bottoms | 1.3778 | 1.076 | 61.3 | 34.0 | 4.7 |
| Plate No. 1 | 1.3350 | 0.998 | 96.7 | 0.8 | 2.5 |
| Plate No. 2 | 1.3338 | 0.998 | 98.0 | 0.0 | 2.0 |
| Plate No. 3 | 1.3343 | 0.995 | 95.5 | 0.0 | 4.5 |
| Plate No. 4 | 1.3385 | 0.984 | 90.0 | 0.0 | 10.0 |
| Plate No. 5 | 1.3589 | 0.918 | 52.0 | 0.0 | 48.0 |
| Plate No. 6 | 1.3636 | 0.854 | 24.6 | 0.0 | 75.4 |
| Plate No. 7 | 1.3636 | 0.833 | 16.5 | 0.0 | 83.5 |
| Plate No. 8 | 1.3632 | 0.823 | 13.0 | 0.0 | 87.0 |

TABLE 6

RUN 5

| sample | Refractive Index | Density | Water | Composition (Percent) | |
|-------------|---------------------|---------|-------|-----------------------|---------------|
| | | | | Glycerin | Ethyl-Alcohol |
| Feed | 1.3764 | 0.974 | 44.0 | 20.0 | 36.0 |
| Bottoms | 1.3812 | 1.075 | 59.0 | 35.0 | 6.0 |
| Plate No. 1 | 1.3520 | 0.956 | 70.0 | 1.0 | 29.0 |
| Plate No. 2 | 1.3624 | 0.884 | 36.5 | 0.5 | 63.0 |
| Plate No. 3 | 1.3632 | 0.854 | 25.0 | 0.0 | 75.0 |
| Plate No. 4 | 1.3635 | 0.838 | 18.5 | 0.0 | 81.5 |
| Plate No. 5 | 1.3632 | 0.831 | 16.0 | 0.0 | 84.0 |
| Plate No. 6 | 1.3630 | 0.823 | 13.0 | 0.0 | 87.0 |
| Plate No. 7 | 1.3629 | 0.819 | 11.8 | 0.0 | 88.2 |
| Plate No. 8 | 1.3628 | 0.815 | 10.0 | 0.0 | 90.0 |

TABLE 7

RUN 6

| Sample | Refractive Index | Density | Water | Composition (Percent) | |
|-------------|---------------------|---------|-------|-----------------------|---------------|
| | | | | Glycerin | Ethyl-Alcohol |
| Feed | 1.3840 | 0.925 | 19.0 | 22.5 | 58.5 |
| Bottoms | 1.4184 | 1.117 | 28.0 | 58.5 | 13.5 |
| Plate No. 1 | 1.3643 | 0.857 | 23.5 | 1.5 | 65.0 |
| Plate No. 2 | 1.3637 | 0.839 | 19.3 | trace | 80.7 |
| Plate No. 3 | 1.3633 | 0.830 | 16.0 | 0.0 | 84.0 |
| Plate No. 4 | 1.3633 | 0.825 | 14.0 | 0.0 | 86.0 |
| Plate No. 5 | 1.3632 | 0.821 | 12.5 | 0.0 | 87.5 |
| Plate No. 6 | 1.3631 | 0.818 | 11.5 | 0.0 | 88.5 |
| Plate No. 7 | 1.3629 | 0.815 | 9.7 | 0.0 | 90.3 |
| Plate No. 8 | 1.3628 | 0.812 | 8.6 | 0.0 | 91.4 |

TABLE 8

RUN 7

Composition (Percent)

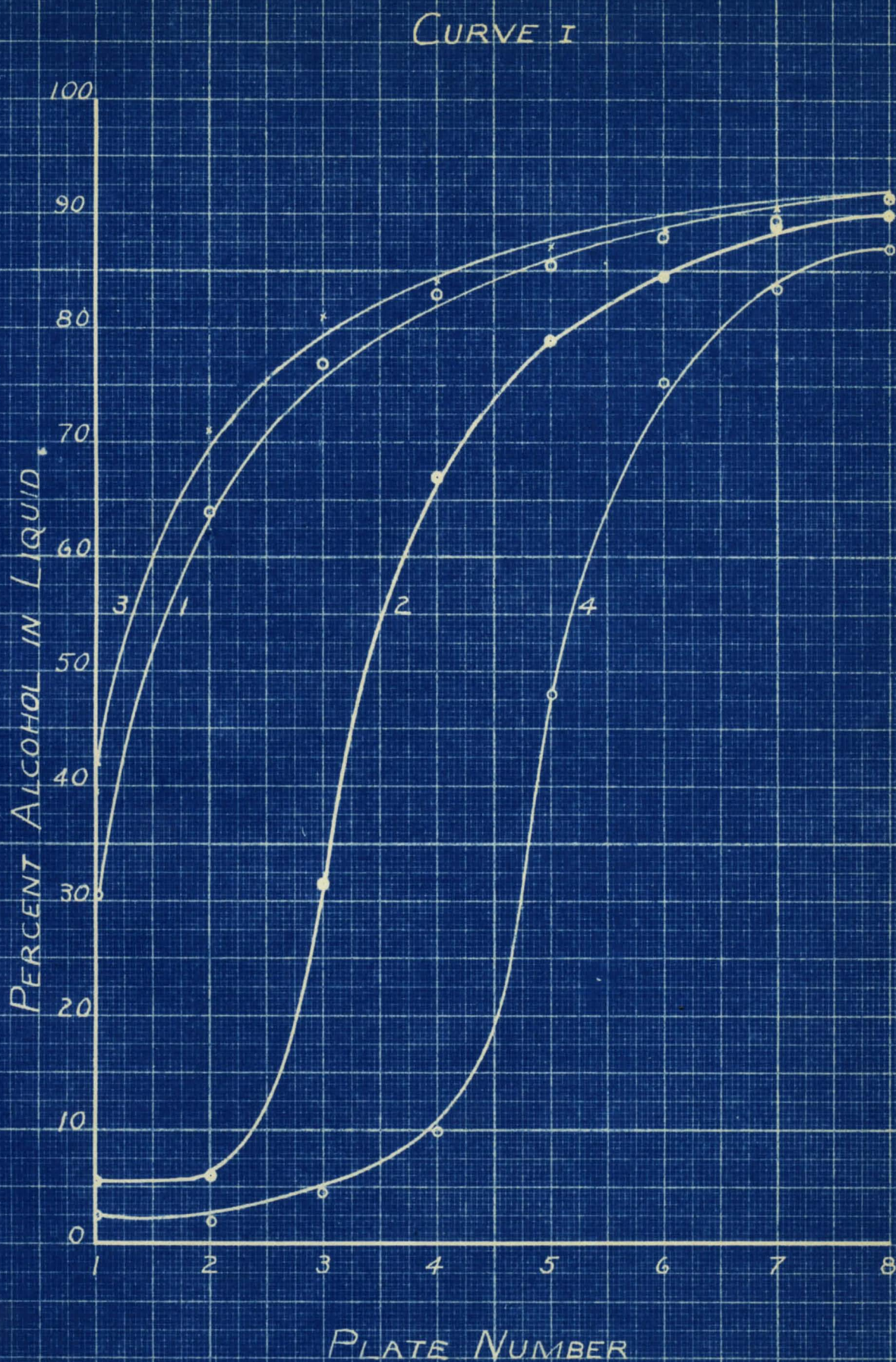
| Sample | Refractive Index | Density | Water | Glycerin | Ethyl-Alcohol |
|-------------|------------------|---------|-------|----------|---------------|
| Feed | 1.3513 | 1.004 | 80.0 | 10.2 | 9.8 |
| Bottoms | 1.3515 | 1.034 | 74.0 | 15.0 | 1.0 |
| Plate No. 1 | 1.3335 | 0.995 | 96.0 | 0.0 | 4.0 |
| Plate No. 2 | 1.3335 | 0.996 | 97.0 | 0.0 | 3.0 |
| Plate No. 3 | 1.3337 | 0.997 | 97.5 | 0.0 | 2.5 |
| Plate No. 4 | 1.3338 | 0.997 | 97.5 | 0.0 | 2.5 |
| Plate No. 5 | 1.3353 | 0.992 | 94.0 | 0.0 | 6.0 |
| Plate No. 6 | 1.3530 | 0.949 | 68.0 | 0.0 | 32.0 |
| Plate No. 7 | 1.3630 | 0.868 | 30.0 | 0.0 | 70.0 |
| Plate No. 8 | 1.3634 | 0.841 | 19.5 | 0.0 | 80.5 |

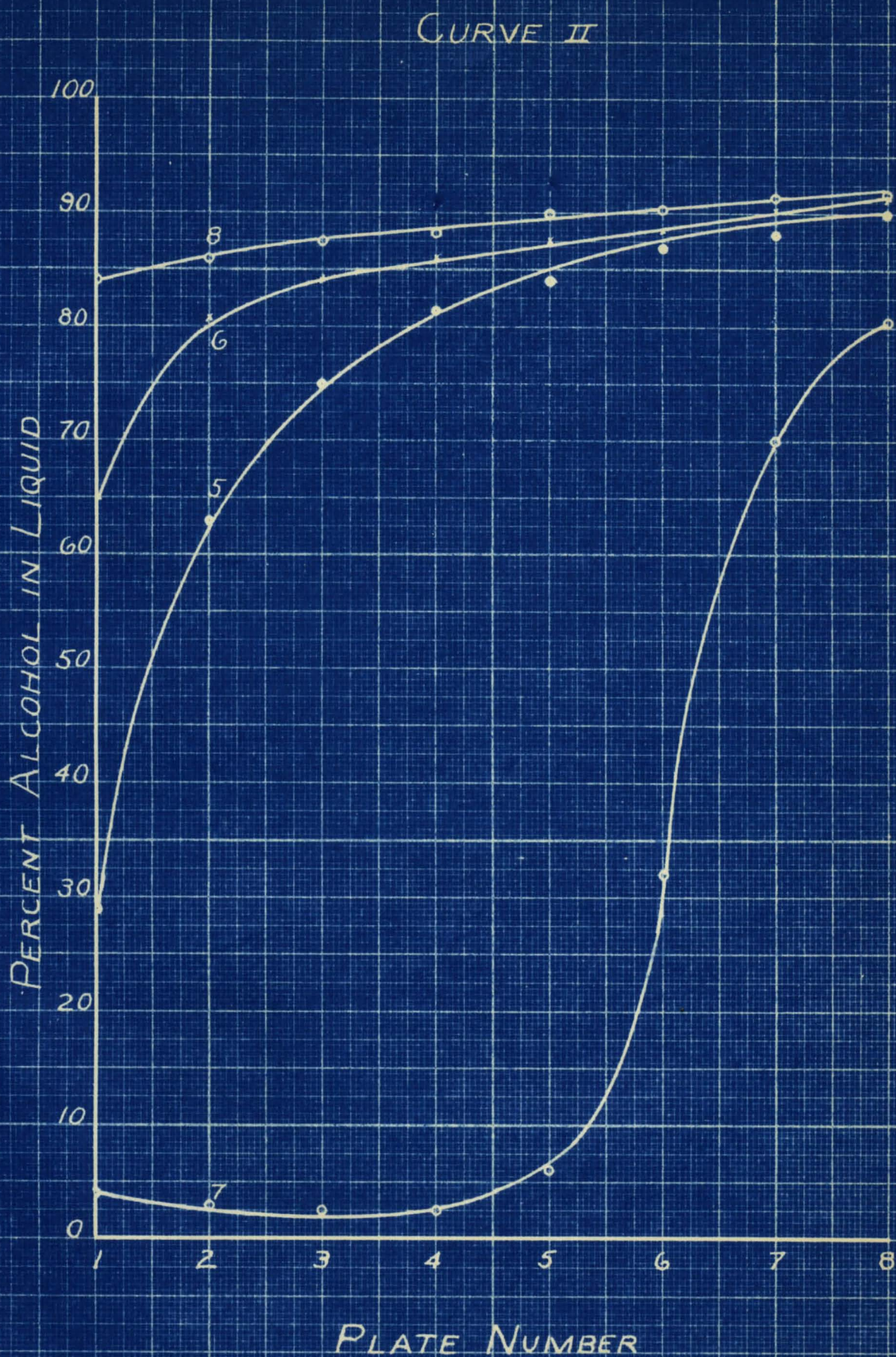
TABLE 9

RUN 8

Composition (Percent)

| Sample | Refractive Index | Density | Water | Glycerin | Ethyl-Alcohol |
|-------------|------------------|---------|-------|----------|---------------|
| Feed | 1.3737 | 0.860 | 10.0 | 12.0 | 78.0 |
| Bottoms | 1.3841 | 0.936 | 23.0 | 23.0 | 54.0 |
| Plate No. 1 | 1.3638 | 0.829 | 16.0 | trace | 84.0 |
| Plate No. 2 | 1.3635 | 0.825 | 14.0 | trace | 86.0 |
| Plate No. 3 | 1.3632 | 0.821 | 12.5 | 0.0 | 87.5 |
| Plate No. 4 | 1.3632 | 0.819 | 11.8 | 0.0 | 88.2 |
| Plate No. 5 | 1.3630 | 0.816 | 10.0 | 0.0 | 90.0 |
| Plate No. 6 | 1.3630 | 0.815 | 9.7 | 0.0 | 90.3 |
| Plate No. 7 | 1.3628 | 0.812 | 8.6 | 0.0 | 91.4 |
| Plate No. 8 | 1.3627 | 0.811 | 8.3 | 0.0 | 91.7 |





Curves 1 and 2 are plots of the percent alcohol in the liquid on each plate versus the plate number for each run. Runs 2, 4, and 7 do not show as high a plate efficiency and as good fractionation when the percent alcohol on the plate is below 35%. The plate efficiency increases as the percent alcohol increases. These curves show a decrease in slope as the alcohol content increases as is to be expected from the equilibrium diagram for the binary system ethyl alcohol-water.

The curves for the other runs show a decrease in slope as the alcohol content in the liquid increases. This means that the degree of separation obtained diminishes as the percent alcohol increases. These curves approach the constant boiling mixture of ethyl alcohol-water as an asymptote.

Table 10 shows the number of plates necessary to get the same separation as may be obtained in an Othmer apparatus. The values range from 1 in the case of run 5, and 6 in the case of run 6.

TABLE 10

| Run No. | Residue Composition (Percent) | | | Comparable Point on Fig. 1 | Percent Alcohol in Vapor Fig. 1 | Number of Plates to Secure Comparable Separation |
|-----------|-------------------------------|----------|---------------|----------------------------------|--|--|
| | Water | Glycerin | Ethyl-Alcohol | | | |
| Run No. 1 | 21.0 | 77.5 | 1.5 | 23 | 72 | 3 |
| Run No. 2 | 44.0 | 55.0 | 1.0 | 9 | 62 | 4 |
| Run No. 3 | 20.0 | 77.5 | 2.5 | 23 | 72 | 2 |
| Run No. 4 | 61.3 | 34.0 | 4.7 | 3 | 63 | 6 |
| Run No. 5 | 59.0 | 35.0 | 6.0 | 10 | 17 | 1 |
| Run No. 6 | 28.0 | 58.5 | 13.5 | 19 | 80 | 2 |
| Run No. 7 | 74.0 | 15.0 | 1.0 | 11 | 3 | 2 |
| Run No. 8 | 23.0 | 23.0 | 54.0 | 16 | 86 | 2 |

CHAPTER VIII

SUMMARY

SUMMARY

Charges of various composition of the ternary system, glycerin-ethanol-water, were subjected to distillation in a semi-plant still. Samples of the liquid on each plate, and of the residue were taken and analyzed by means of density and refractive index. The data and results are shown in tables II thru IX inclusive.

CHAPTER IX

CONCLUSIONS

CONCLUSIONS

The ternary mixture glycerin-ethyl alcohol-water can be separated by distillation. The alcohol can be recovered by the use of a rectifying column as 89.4 mol percent alcohol. The glycerin can be obtained in a practically pure state by vacuum distillation.

The analysis of the liquid in the kettle and on the first plate shows that the separation obtained in this step approximates very closely the data obtained by Watkins (1) in an Othmer apparatus. The remaining plates in column act as in the rectification of a binary mixture.

If the process were continuous and the liquid were fed on one of the plates, the exhausting column would give results similar to those obtained by Watkins (1) in an Othmer apparatus, while the rectifying column would function as in the distillation of a binary mixture.

An increase in the percent of glycerin in the kettle increases the alcohol content of the liquid on the first plate and consequently the percent alcohol in the liquid on each succeeding plate.

This condition is caused by the increase in boiling point of the liquid in the kettle induced by the increase in the percent of glycerin. This elevated boiling point gives a superheating effect.

No quantitative statement can be made concerning the number of plates required to give the same separation as the Othmer apparatus because the plate efficiency according to Underwood (8) varies with the composition of the feed. The number varied from 1 to 6, the value 2 occurring four times. (See table 10).

CHAPTER X

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